

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No. : 10/580,699 Confirmation No. 5404  
Applicants : Shin MASAOKA et al.  
Filed : May 25, 2006  
Title : Process for Producing Phosphonium Borate Compound,  
Novel Phosphonium Borate Compound, and Method of  
Using the Same  
Group Art Unit : 1621  
Examiner : Chukwuma O. Nwaonicha  
Customer No. : 28289

Commissioner for Patents  
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**DECLARATION UNDER 37 C.F.R. § 1.132**

I, Shin MASAOKA, declare and say as follows:

1. I graduated from the Graduate School of Engineering, Hiroshima University in March, 1996. Since April, 1996, I have been an employee of Hokko Chemical Industry Co., Ltd., and till the present time, I have been engaged in the Fine Chemicals Process Research Department.

2. I am familiar with the subject matter of U.S. Application Serial No. 10/580,699, of which I am an inventor. Also, I am familiar with the rejection of claims 1-3 under 35 U.S.C. §102(b) as being anticipated by Smith et al., "Phosphorus mustards. III. Bis(2-chloroethyl)methylphosphine oxide and bis(2-benzoxyethyl)methylphosphine", J. Med. Chem., 11(5), 1060-3 (Sept. 1968); claims 1-3 under 35 U.S.C. §102(b) as being anticipated by Aylion et al., "Proton Transfer in Aminocyclopentadienyl Ruthenium Hydride Complexes", Organometallics, 18(20), 3981-3990 (1999); and claims 1-6 under 35 U.S.C. §103(a) as being unpatentable over Smith et al. or Aylion et al. in the '699 application.

3. The following experiment was conducted either by myself or under my direct supervision. This experiment demonstrates that a yield of tri-tert-butylphosphonium tetraphenylborate:  $(\text{tert-Bu})_3\text{PH}\cdot\text{BPh}_4$  by a process including the concentration step according to Smith et al. is 66%.

[Comparative Example]

Production of tri-tert-butylphosphonium tetraphenylborate

A 300-ml four-necked flask sufficiently purged with argon was equipped with a stirrer, a thermometer and a reflux condenser. 8.1 g (40 mmol) of tri-tert-butylphosphine and 130 ml of chloroform were weighed in the flask, followed by stirring to dissolve tri-tert-butylphosphine. While the stirring was continuously carried out, hydrochloric acid gas was bubbled through the solution at 0°C for 15 minutes. The reflux condenser was replaced with a distiller. The solution was concentrated at 25°C at 0.1 mmHg to obtain a residue.

A 300-ml four-necked flask was equipped with a stirrer, a thermometer and a reflux condenser. 15.1 g (44 mmol) of sodium tetraphenylborate and 60 ml of methanol were weighed in the flask, followed by stirring to dissolve sodium tetraphenylborate. While the stirring was continuously carried out, the residue was added to the solution, and the mixture was stirred at 25°C for 3 hours. After the completion of the reaction, the precipitated product was filtered off. The so-obtained crystal was suspended in 100 ml of toluene at 50°C, and the suspension was cooled to 25°C and filtered. The product filtered off was washed with 100 ml of toluene. The crystal was then suspended in 100 ml of methanol at 50°C, and the suspension was cooled to 25°C and filtered. The product filtered off was washed with 100 ml of methanol. The crystal obtained was dried to give 13.7 g of objective tri-tert-butylphosphonium tetraphenylborate as white crystal. The yield (mol%) was 66% based on tri-tert-butylphosphine.

The crystal was analyzed by the same methods as indicated in Example 1 of the Specification and was identified to be tri-tert-butylphosphonium tetraphenylborate.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both, under Section 1001 of Title 18 of the

United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Shin MASAOKA

Shin Masaoka

Date October 29, 2009